THE INFLUENCE OF THE CONTACT FORCE IN DILATOMETRY

Force-Controlled Measuring Cells in dilatometry

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ABSTRACT

This article presents the design and construction of a force-controlled measuring cell in dilatometry. The proposed system is designed for measurement and control of the contact force in dilatometer measurements in order to minimize its effect on the length signal and expand the application range of dilatometry for measurement of visco-elastic material properties.

Index Terms – dilatometry, DIL, thermal expansion, thermal analysis, CTE

1. INTRODUCTION

Exact measurement of dimensional changes is important in modern material research, e.g., analysis in the field of zero-expansion materials, polymers, foams, sandwich materials and ceramics. The length changes of a sample as a function of temperature can be measured with common dilatometers under a small load as defined in national standards [1], [2]. Especially for applications with soft materials, this contact force – generated by the precision-engineered measuring cell – influences dilatation to an extent that can no longer be ignored and which is crucial for exact determination of the thermal expansion and CTE (coefficient of thermal expansion).
The thermal expansion $\propto (T)$ and CTE $\propto (\Delta T)$ are calculated as follows:

$$\propto (T) = \frac{1}{L_0} \cdot \frac{dL}{dT} \quad (1)$$

$$\propto (\Delta T) = \frac{1}{L_0} \cdot \frac{\Delta L}{\Delta T} \quad (2)$$

L	*measured length change of the sample*
L_0	*initial length of the sample*
T	*measured sample temperature*

Figure 1 describes the principle of a dilatometer where a sample is inserted into a special sample holder. The sample is positioned directly against an arrester on the one side and against a pushrod on the other, which transmits the length change to a linear variable differential transducer (LVDT). The whole arrangement is placed in a moveable furnace to apply a programmed temperature.

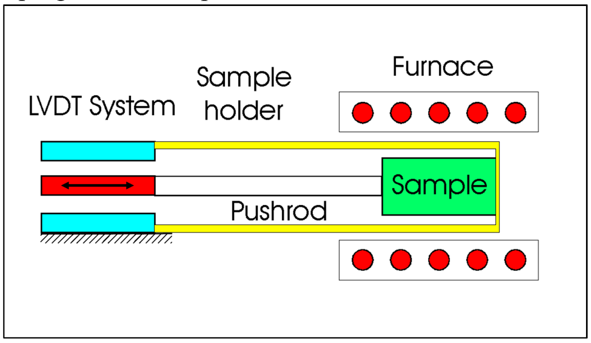

Figure 1: Principle of a dilatometer measurement [3]

As the sample length changes during the temperature program, a calibrated output signal proportional to the displacement is recorded (see point 3.1.1). The temperature of the heating element of the furnace and of the sample is detected by thermocouples.
As the sample holder and the front part of the pushrod are affected by the same temperature program like the sample, their dimensions are also changing. Therefore, not only the length changes of the sample and sample holder but also of the pushrod have an effect on the measurement of the displacement. This is the reason why the dilatometer curve needs to be corrected either by tabulated expansion data of the used sample holder and pushrod material or by a correction curve measured with a known standard material to eliminate systematic errors. This correction of the raw dilatometer data is necessary to gain objectives results of the sample behavior [3], [4].

2. LIMITATIONS OF DILATOMETERS

Common dilatometers are designed with a linearly guided pushrod which is in contact with the sample and transfers the length change of the sample to the measuring cell. This dimensional change is detected by a position sensor with a resolution in the nm-range

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(e.g. LVDT). The contact force is applied and can be manually adjusted within a small range (0.15 N – 0.45 N) by changing the preload of a spring (see Figure 2). This design in combination with the spring rate is the reason why the contact force changes during the measurement due to the expansion of the sample and this change might even be non-linear. In the current state of dilatometry, detection of the contact force is not common. Without a design change, this would only be possible by detecting the elongation of the spring based on the predetermined spring rate. Furthermore, contact forces are generally too high and the adjustment range is too small for analyzing compressible materials, e.g. foam, insulation material or green bodies. Additionally, only static contact forces are possible and thus no evaluation of visco-elastic properties can be made.

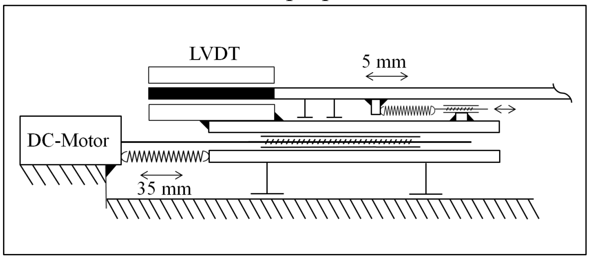

Figure 2: Technical principle of a dilatometer measuring cell

Guidance of the pushrod realized by standard linear guides itself is a source of errors. Sliding and rolling friction as well as stick-slip-effects can impact the thermal expansion measurements and cause deviation as well as jumps in the length signal.

Another limitation of common dilatometers is that the whole array of the sample length up to 50 mm cannot be measured with one pushrod. Due to a historically given limitation of the travel length of approx. 35 mm, two different long pushrods with a different length and of course differing properties needed to be used (see Figure 2).

A further impact on the measuring accuracy of dilatometry is that the initial length of the sample is determined manually by using a caliper or micrometer gauge. Despite the inferior accuracy of this equipment, the initial length of the sample is not detected with the later used contact force and leads to a deviating L_0. This effect of measuring the initial sample length manually with unknown forces becomes especially evident with soft materials like foams which additionally get crimped by the pushrod and this reduction of the length is even not detected.

The established analog measuring principle of an LVDT (linear variable differential transducer) has the disadvantage that a long measuring range and high resolution are conflictive targets and also, that a calibration is needed. To achieve a resolution in the nm-range, a maximum measuring range of about 5 mm can be realized with a 24-bit ADC, though the electronic signal processing is sensitive to temperature changes.

3. APPROACH FOR A FORCE-CONTROLLED MEASURING CELL

In this article, reduction of the contact force on the length signal will be discussed and the possibility of expanding the measuring range of common dilatometers will be addressed. A new design of a vacuum-proof and thermally stabilized measuring cell which is able to detect and control the contact force will thus be described. Furthermore, a design which is independent of any friction and grease effects will be discussed but also function-integrated parts and constructions. A position sensor technology appropriate for expanding the common measuring range and resolution will be reviewed. Analysis investigating different principles of linear actuators will highlight the suitability of applying the sample with constant and modulated forces.

3.1 Functional components

3.1.1. Length sensors

According to national standards, the length sensor detecting the length change of a sample needs to have a resolution better than 0.1 % and a proven linearity of at least ±0.1 % of its measuring range [3] as well as an error limit of 0.5 µm [5]. Following the request for a resolution in the nm-range, the length measuring system of an LVDT is suitable. While in the past, incremental linear encoders with digital output based on a patterned scale had a much lower resolution compared to LVDTs, recent development of interpolation technologies reach a comparable resolution to LVDTs [6], [7]. Therefore, these two different technologies detecting the length changes are tested and compared with special emphasis on the thermal behavior.

A major difference is the calibration where the LVDT is commonly calibrated by using a micrometer gauge to get the correlation of the voltage output and displacement. The quality of the calibration is strongly determined by the used length measurement device. Due to inherent instabilities, recalibration needs to be done frequently. Besides this manual calibration process, LVDTs show nonlinearities. Main causes are winding irregularities in the coils and noise in the signal processes. These influences are proportional to the measuring range.

In contrast, the calibration and linearity of an incremental linear encoder and its scale is set during the manufacturing process of the producer where lines are photo engraved perennially. Therefore, sources of defects are reduced, leading to a higher level of accuracy. If necessary, incremental linear systems can be calibrated with an interferometer.

The different length measurement technologies described above are compared by means of a dilatometer measurement on a 35 mm Al_2O_3 sample from room temperature to 500 °C by simultaneous detection of the length signal at the end of the pushrod (see Figure 3, measurement done with a NETZSCH DIL402C). The sample holder and pushrod are made of Al_2O_3.

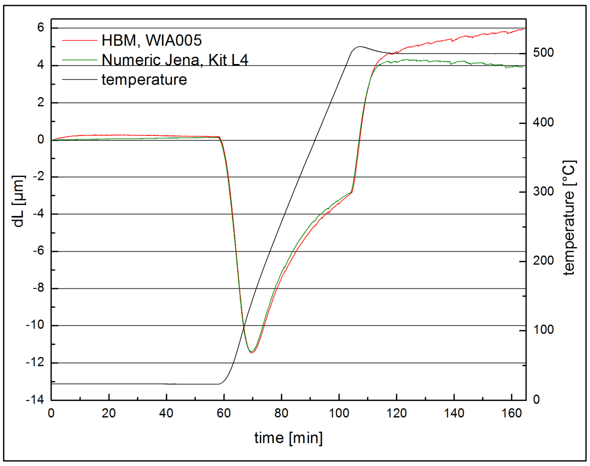

Figure 3: Influence of the sample-temperature on a LVDT (HBM, WIA005) compared to an incremental linear encoder (Numeric Jena, Kit L4)

Figure 3 shows a low drift of the LVDT compared to a negligible deviation of the incremental linear encoder for 60 minutes at room temperature. During the temperature change from room temperature to 500 °C, both measuring systems detecting the length change of the sample, sample holder and pushrod show almost the same behavior. In contrast, the LVDT shows a significant drift of the length signal at 500 °C sample temperature compared to the digital encoder. It can be seen that the LVDT is more sensitive to temperature changes compared to the digital encoder with a patterned glass-scale which is minimally temperature-sensitive and therefore requires no special environmental conditions (see Figure 3).

In summary, an incremental linear encoder is more suited for detecting the length change of materials influenced by temperature changes. Furthermore, an incremental linear encoder allows an extension of the measuring range over the whole sample range up to 50 mm without an effect on its high resolution. This enables simultaneous detection of the initial length with a low temperature dependence. Additionally, accuracy, resolution, and linearity of an incremental linear encoder do not change and need to be maintained.

3.1.2. Linear actuator

In order to apply the exact predetermined contact force, the pushrod needs to be driven by an actuator allowing forces up to about 3 N with a control accuracy of about 1 mN. Contrary to most applications, the actuator needs to generate a certain force independent of its actual position. So a detection and a control circuit of the actual contact force is necessary (see 4.1).

With possible stroke lengths up to 100 mm, linear DC-servomotors and piezo-drives are suited for this application.

In the following, a comparison between these two actuators is made based on tests. Therefore, an experimental setup was designed where the actuators contact a guided measuring cell. The position of the measuring cell was changed by a spindle drive with a velocity up to 20 µm/s in order to simulate a thermal length change of the sample. A force-controlled electronic control circle (see 4.1) enabled the actuators to maintain the predetermined contact force.

Figure 4 shows the ability of the two different actuator principles to apply constant forces at changing positions.

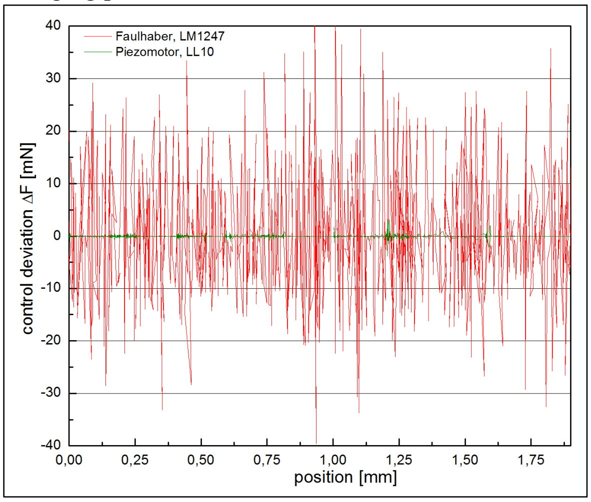

Figure 4: Control deviation of a linear DC-servo motor (Faulhaber, LM1247) [8] compared to a piezo-drive (Piezomotor, LL10) [9]

The test shows that the linear DC-servomotor achieves a force noise of about 30 mN. This actuator is based on a continuous current linear motor principle with a changing pole movable rod [6]. Advantages of such an actuator are an extendable rod and high possible dynamics without reset forces [8].

In a static operation, the induction characteristic is linear to the applied contact force solely in the middle area and shows a significant drop at the area of the commutation. This leads to a non-linear force/distance and force/current characteristic thus affecting the control parameters and properties of the force control in dependence of the commutation [6]. In this application with extremely slow and narrow distance changes, this principle leads to oscillation and thus noise of the contact force which is not acceptable (see Figure 4).

In contrast, the piezo-actuator using the piezoelectric-effect and operating with a non-resonant principle of piezo-legs walking on a ceramic rod achieves a force noise of less than 1 mN (see Figure 4). The contact between the drive legs and the drive rod is realized with frictional connection. Because of more than one leg always being in contact with the rod, this actuator is free of clearance and reset forces. The position is maintained even if the power is switched off [9].

The test shows that a piezo-actuator based on a piezo-leg principle can realize and apply a contact force during a position change with a force control noise smaller than 1 mN. Due to the extremely small step size and a holding force given by the friction-based working principle, this actuator is suited for a force-controlled measuring cell. The disadvantage of changing step size at different applied forces and therefore a changing number of steps per distance can easily be overcome by the electronic control circle.

3.2 Design versions and working principles

The central function of a force-controlled measuring cell is to analyze the correlation of the temperature and elongation of a sample. This function can be subdivided in different function elements which are linked together as shown in Figure 5.

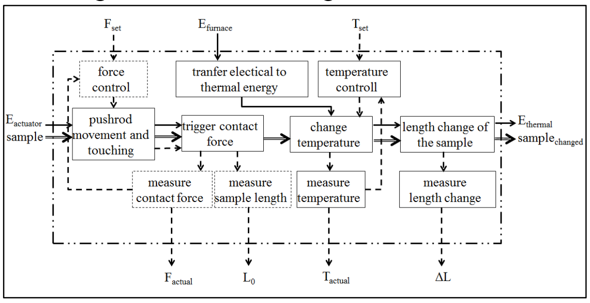

Figure 5: Functional structure of a force-controlled dilatometer measuring cell

The moveable guided pushrod touches the sample with a predefined contact force, which is detected by a force sensor and adjusted with a controller. The closed-loop control regulates the actuator which generates the contact force and transfers it through the pushrod to the sample. The actuator and its control need to be carefully designed to avoid any oscillations.

The controlled temperature change of the sample leads to an elongation which is detected by a displacement measurement system. Due to the sample with a possible length up to 50 mm, movement of the already described functional parts is necessary. This movable measurement unit consists of the guidance of the pushrod, measurement, excitation and absorption of the contact force as well as the measurement of the elongation. This axial movement of the measurement unit likewise requires guidance, an actuator as well as a displacement sensor to detect the initial sample length.

Figure 6 shows the described necessary functions and components to realize a force-controlled measuring cell for dilatometers.

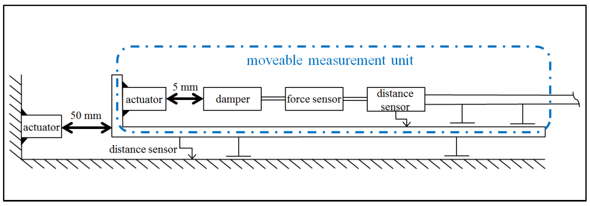

Figure 6: General schematic of a force-controlled dilatometer measuring cell

The alignment of the components shown and the function structure serve as a basic principle for the development of possible realization solutions and technical principles.

Development of sub solutions, different technical principles and evaluation of the technical principles by technical criteria – not explicitly described in this article, led to a priority technical principle as shown in Figure 7.

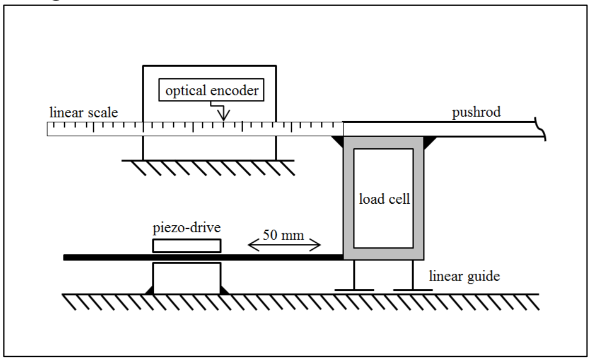

Figure 7: Technical principle of a force-controlled measuring cell for dilatometers

Due to the results of 3.1, a technical principle containing the components of an incremental linear encoder (3.1.1) and an actuator based on piezo-technology (3.1.2) is preferred and will be further described in the next points.

3.3 Basic design of the favorite technical principle

Figure 8 shows the mechanical design of the preferred technical principle. The functions of measuring the length change and initial sample length are executed by an incremental linear encoder system containing a patterned scale and an optical encoder. The contact force is detected by a load cell and applied by a piezo-drive. The movable components (load cell, patterned scale and pushrod) are guided by a linear guide. The whole system is assembled to a base frame (see Figure 8)

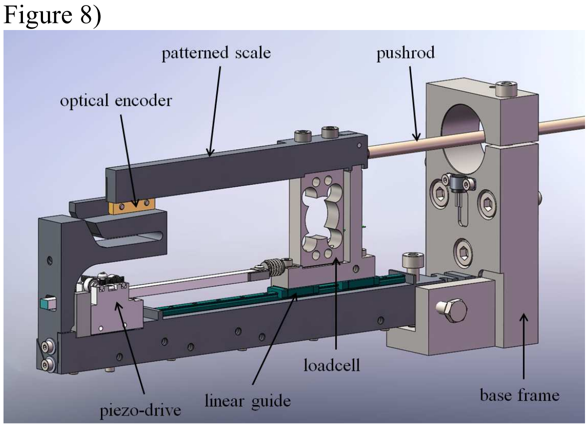

Figure 8: 3-D-drawing of a force-controlled measuring cell

3.4 Design rules

Harmful failure impacts on the whole function are eliminated by use of the design rules of the reduction of failures [6].

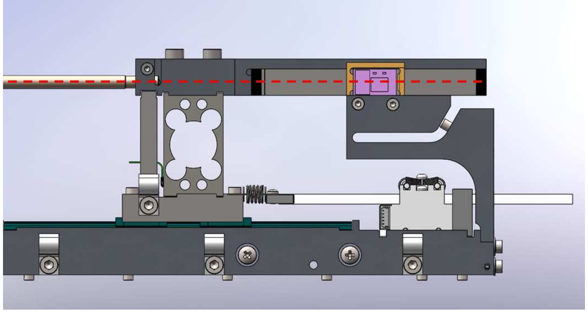

Figure 9: Abbe comparator principle to avoid errors of first order [6]

Invariant or innocent structures have the advantage that no or only errors of second or higher orders emerge. Such characteristics can be realized with the deployment of the Abbe comparator principle. Due to a straight axial alignment and transmission of the elongation of the sample, over the pushrod to the displacement measuring system, errors of first order are prevented [6] (see Figure 9, red dashed line).

The principle of function integration describes an arrangement of one or more components, which does not only realize a single sub-function but is involved in several sub-functions by a built-in maximum utilization [6]. The components for excitation of the contact force also carry out the sub-function to realize the axial movement of the measurement unit emerged by the different sample lengths (see Figure 10, blue ellipse). The piezo-actuator, tested in 3.1.2, is able to perform both functions integrated because of its long stroke length compared to other actuators such as a voice-coil-actuator.

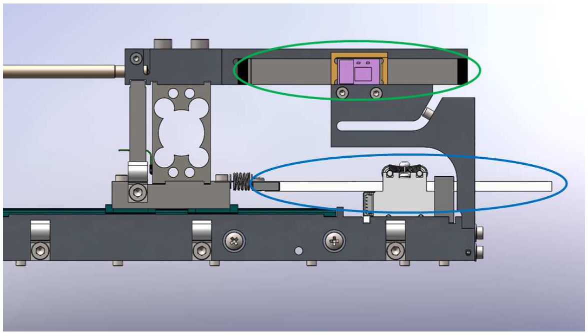

Figure 10: Principle of function integration: incremental linear encoder and piezo-drive each executing two sub-functions

For measurement of the initial sample length and the expansion of the sample (see Figure 6, point 3.2), normally two independent displacement measurement systems are needed. The incremental linear encoder, described under 3.1.1 with a patterned scale and an optical encoder can fulfill the requirements of both sub-functions, a wide measuring range for the detection of the initial sample length, and an extremely high resolution in a lower measuring range for measuring the length change of the sample (see Figure 10, green ellipse).

With this principle, a reduction of numbers of components hence a saving of installation and adjustment effort as well as simplification of the apparatus structure and a more intense material utilization can be achieved.

Over-determined designs and their constrained symptoms can be controlled through the following methods [6]:

Constructive schemes in order to reduce the influence of over-constraints as elimination is not reasonable. In the present design, the coupling of the piezo-actuator to the guide is realized in the form of a bending beam axial preloaded by a compression spring. This combination shows a high stiffness in the push direction while being weak in all other directions. In this way, differences in height and angle deviations between the two guided elements are balanced (see Figure 11, green ellipse).

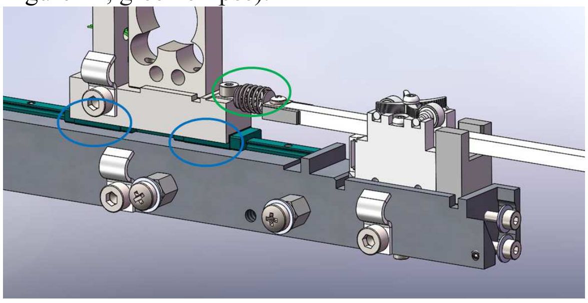

Figure 11: Reduction of the influence of over-constraints with a bending beam (green) and a bedstop (blue)

Furthermore, over-determinations can be restrained by technological methods. Production of identical dimensions of length and angle as well as correspondingly close manufacturing tolerances control over-constraints and their impacts. In the present design, the guide adapter is assembled on two guiding carriages of the linear guide by a shared bedstop on the side. Furthermore, manufacturing tolerances for dimensions- and flatness-deviations are tolerated in the 0.01 mm range (see Figure 11, blue ellipses).

3.5 Adjustments

Functional testing and the tolerances given by the manufacturer of the incremental linear encoder indicate that adjustment of the position of the optical encoder to the scale is needed. Especially the rotation around the z-direction ($\Delta\varphi_z$) and the displacement in the y-direction (Δy) are critical for the quality of the displacement signal in the x-direction. To overcome these issues, the mechanical design of the measuring cell includes adjustment means to set a definite position of the optical encoder to the patterned scale (see Figure 12).

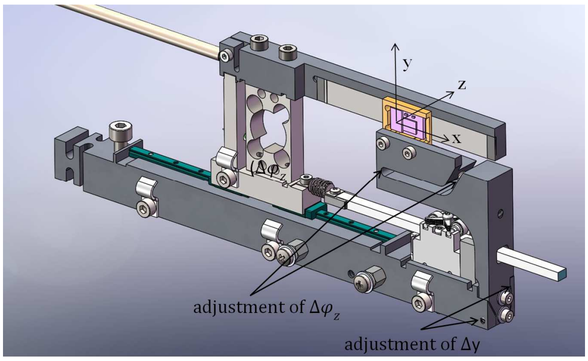

Figure 12: Adjustments to set a definite position of the optical encoder to the patterned scale

4. ELECTRONIC CONTROL

4.1 Control structure

As discussed in 3.1.2 and 3.2, the actuator applying the contact force on the pushrod, thus being transferred to the sample, is controlled by the contact force (see Figure 13).

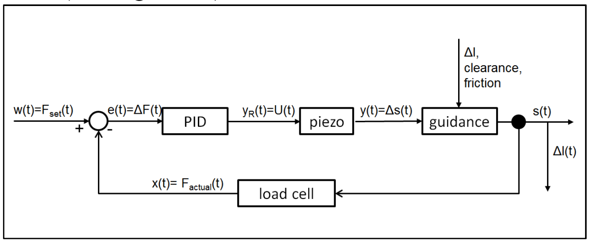

Figure 13: Electronic control circle

The contact force is set by the user which is compared with the actual force detected by the load cell. The PID-control realized by a microcontroller calculates and changes dependent on the deviation of the force an output for the piezo-actuator. This actuator moves its stroke simply forward or backward thus changing the position of the guidance until the defined contact force is achieved. Independent of the control circle of the contact force, the length change of the measuring unit affected by the elongation of the sample is detected.

4.2 Force calibration

Changes in contact force influence the length signal due to the effect that every component and its material involved in force transmission is elastic and has its own specific stiffness. Furthermore, the kinematics of the parallel-spring-guide thus influencing the position of the incremental linear optical encoder affects this effect. First measurements without a sample (baseline dilatometer curve) show an increasing length change of a few µm with a rising contact force up to 3 N under isothermal conditions (see Figure 14).

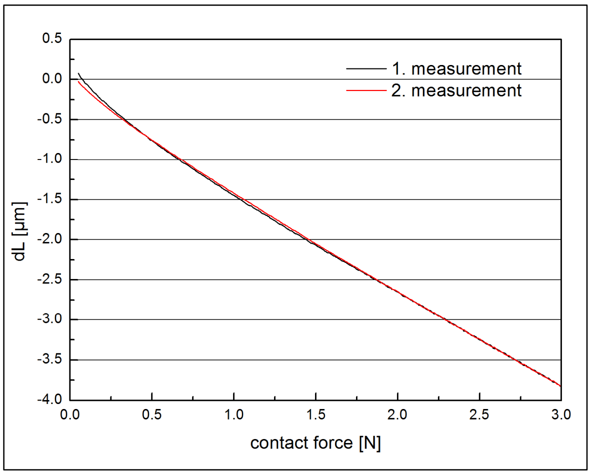

Figure 14: Length signal as a function of the contact force

In order to overcome the effects of changing forces on the length signal, a new approach is proposed, where the effects of this measuring cell stiffness in the measurements are computationally corrected. For this, a model including all dependencies like different forces, initial sample lengths and temperatures needs to be implemented.

5. EXPERIMENTAL RESULTS

After assembly of a new measuring cell, first experimental measurements were carried out. These measurements were performed using external electronic control systems like an evaluation-board for the incremental linear encoder and an external driver electronic for the piezo-actuator in order to have a first qualitative proof of the system's functionality.

The first experiment shows the influence of the contact force for the determination of the initial length of the sample L_0. Therefore, the length of an insulation material sample (Styrodur®, BASF) was multiply measured manually in comparison to the automatic measurement with the newly designed measuring cell. Figure 15 shows that the initial sample length is strongly dependent on the contact force. With contact forces of a few mN up to 3 N, the initial sample length can vary up to 5 %. The shaded area marks the manually measured initial length average and its standard deviation. This procedure corresponds to an automatic measurement with contact forces in the range of approximately 0.85 mN to 1,05 mN. Especially with contact forces between 0.1 mN and 1 N, the automatic mode recording of L_0 shows an explicitly lower standard deviation in comparison to the manual measurement.

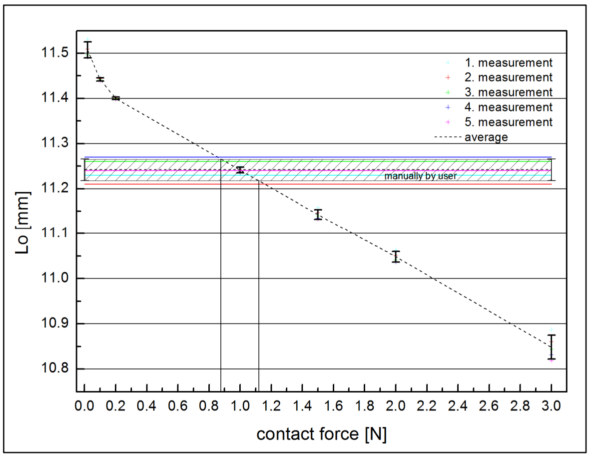

Figure 15: Determination of the initial sample length L_0 with different contact forces (Styrodur®, BASF)

Dilatometer measurements from room temperature to 65 °C on a packaging foam show the influence of different small contact forces on the CTE (coefficient of thermal expansion), as can be seen in Figure 16. With increasing temperature, the material begins to expand and is followed by a shrinkage where the sample gets soft. The measurements in Figure 16 demonstrate that the magnitude and start temperature of the sample shrinkage are extremely dependent on the contact force.

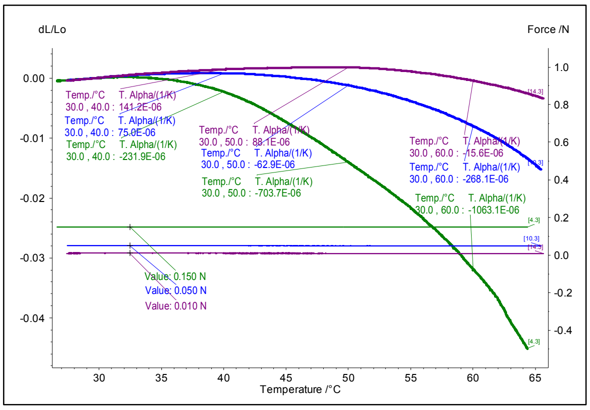

Figure 16: Influence of different contact forces on the thermal expansion of a soft sample

A force-controlled measuring cell enables to test creeping effects of materials as shown in Figure 17 on a sample of Styrodur® (BASF). A force step from 10 mN to 3 N shows an immediate decrease in sample length of about 0.4 mm. The constant force of 3 N for approximately 32 min leads to a retardation of about 0.1 mm.

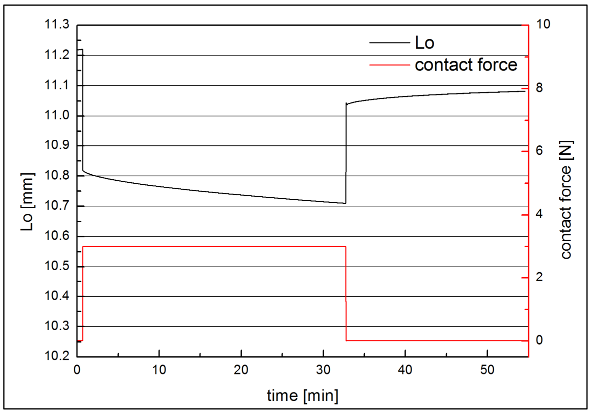

Figure 17: Creeping effect of an insulation material (Styrodur®, BASF) after changing the contact force from 10 mN to 3 N

Furthermore, the Young's modulus can be determined by using the ratio of a force to length change as shown in Figure 17:

$$E = \frac{\sigma}{\varepsilon} = \frac{\Delta F}{A} \cdot \frac{L_0}{L} \qquad (3)$$

A	*cross-section area of the sample*
L_0	*initial length of the sample*
ΔF	*measured force change of the pushrod*
L	*measured length change of the sample*

6. CONCLUSION

A novel measuring cell for dilatometers was proposed and implemented. The system was conceived for detecting the initial length of a sample and its length changes due to a controlled temperature program. Furthermore, the new measuring cell is able to detect and control the contact force in order to reduce measurement failures and impacts on the length signal. The function integrated construction allows equivalent measurement of the initial sample length and the length change by the same component.

Furthermore, this length change is measured with a constant high resolution in the nm-range over the whole measuring range of about 50 mm. Thus, the technology of an incremental linear encoder and a piezo-drive make the use of two different pushrods redundant.

The constructed measuring cell's functionality was demonstrated by means of several experimental measurements on different samples as can be seen in Figure 15 to Figure 17.

In order to overcome the effects of measuring the initial sample length with deviating contact forces, the new system provides a solution to automatically detect the initial length using a predefined contact force with a possible resolution of 1 mN.

With the proposed measuring cell, the application field of dilatometry can be further expanded to the measurement of visco-elastic material properties like creeping effects and others, as can be seen in Figure 17.

7. ACKNOWLEDGMENTS

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